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# Collected By:

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Report Date: 2019-12-04 Final Report

# South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id: 20191113-003

Lab Sample Id: 19PE008836

Customer Sample Id: North Lagoon - AltEn

Sample Description: WS C WW - water

Date Collected: 2019-11-12

Date Received: 2019-11-13

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	33.9	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	7070	5	LC-MS/MS	2019-11-21	2019-11-29
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	64.5	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	10.3	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	206	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	40.8	5	LC-MS/MS	2019-11-21	2019-11-21
lpconazole	ppb	181	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	15.1	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	149	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	634	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2450	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	2400	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	36.0	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	ND	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	ND	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	ND	110	ND	ND	ND
Clothianidin	ppb	19PE008837	102	ND	ND	ND
Cyfluthrin 1-4	ppb	ND	110	ND	ND	ND
Cyhalothrin 1-2	ppb	ND	110	ND	ND	ND
Cypermethrin 1-4	ppb	ND	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	ND	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	<10	112	ND	ND	ND
Glyphosate	ppb	188	82.3	ND	ND	ND
Imidacloprid	ppb	19PE008837	108	ND	ND	ND
lpconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	ND	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

#### Definitions:

ppb - parts per billion

Detection Limit - Lowest concentration that can be quantitatively reported with confidence

ND - Not Detected above the limit of quantification

Duplicate - Concentration found in repeat sample analysis

Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample

Matrix Blank - A similar-matrix, blank sample is evaluated

Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

## **BRIEF METHOD DESCRIPTION**

## Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

### Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

# Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

# Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)

Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.

https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

### Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

## Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

## Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

## Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

#### Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

# Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04

# South Dakota Agricultural Laboratories has examined the sample of

Final Report

Limfinite Package Id: 20191113-003

19PE008837 Lab Sample Id:

Customer Sample Id: West Lagoon - AltEn

Sample Description: WS C WW - water

Date Collected: 2019-11-12 Date Received: 2019-11-13

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	111	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	31000	5	LC-MS/MS	2019-12-03	2019-12-03
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	66.2	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	735	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	ND	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	116	10	LC-MS/MS	2019-11-14	2019-11-22
lmidacloprid	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-21
lpconazole	ppb	134	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	150	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	216	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2160	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	24000	5	LC-MS/MS	2019-12-03	2019-12-03
Trifloxystrobin	ppb	53.3	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	ND	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	32400	102	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	ND	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
lmidacloprid	ppb	315	108	ND	ND	ND
lpconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	ND	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Dyraclastrohia	nnh	10000000	105	ND	ND	ND
Pyraclostrobin	ppb	19PE008838	105	טא	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	ND	91.4	ND	ND	ND
Thiamethoxam	ppb	23300	123	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

#### Definitions:

ppb - parts per billion

Detection Limit - Lowest concentration that can be quantitatively reported with confidence

ND - Not Detected above the limit of quantification

Duplicate - Concentration found in repeat sample analysis

Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample

Matrix Blank - A similar-matrix, blank sample is evaluated

Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

# **BRIEF METHOD DESCRIPTION**

## Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

### Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

# Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

# Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)

Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.

https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

### Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

## Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

## Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

## Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

#### Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

# Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 Final Report

# South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id: 20191113-003

Lab Sample Id: 19PE008838

Customer Sample Id: Field Blank (Between North Lagoon and South Lagoon

Sampling) - AltEn

Sample Description: WS C WW - water

Date Collected: 2019-11-12

Date Received: 2019-11-13

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Ipconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22

Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	ND	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	ND	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	124	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	ND	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	ND	119	ND	ND	ND
Difenoconazole	ppb	ND	116	ND	ND	ND
Dimoxystrobin	ppb	ND	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	ND	110	ND	ND	ND
Fluconazole	ppb	ND	124	ND	ND	ND
Fluoxastrobin	ppb	ND	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
lmidacloprid	ppb	19PE008837	108	ND	ND	ND
lpconazole	ppb	ND	103	ND	ND	ND
Isavuconazole	ppb	ND	118	ND	ND	ND
Itraconazole	ppb	ND	130	ND	ND	ND
Metconazole	ppb	ND	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	ND	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	ND	107	ND	ND	ND
Posaconazole	ppb	ND	117	ND	ND	ND
Propiconazole	ppb	ND	116	ND	ND	ND

Prothioconazole	ppb	ND	122	ND	ND	ND
Pyraclostrobin	ppb	ND	105	ND	ND	ND
Ravuconazole	ppb	ND	122	ND	ND	ND
Tebuconazole	ppb	ND	93.5	ND	ND	ND
Tetraconazole	ppb	ND	116	ND	ND	ND
Thiabendazole	ppb	ND	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	ND	96.5	ND	ND	ND
Uniconazole	ppb	ND	122	ND	ND	ND
Voriconazole	ppb	ND	97.9	ND	ND	ND

#### Definitions:

ppb - parts per billion

Detection Limit - Lowest concentration that can be quantitatively reported with confidence

ND - Not Detected above the limit of quantification

Duplicate - Concentration found in repeat sample analysis

Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample Matrix Blank - A similar-matrix, blank sample is evaluated

Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

### **BRIEF METHOD DESCRIPTION**

## Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

## Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

# Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

## Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

## Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

## Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)

Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.

https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

#### Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

### Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

## Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

#### Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS

L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

### Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 Final Report

# South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id: 20191113-003

Lab Sample Id: 19PE008839

Customer Sample Id: North Lagoon Duplicate Sample AltEn

Sample Description: WS C WW - water

Date Collected: 2019-11-12

Date Received: 2019-11-13

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	32.4	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	5980	5	LC-MS/MS	2019-11-21	2019-11-29
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	61.7	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	312	5	LC-MS/MS	2019-11-21	2019-11-22
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-14	2019-11-22
Glyphosate	ppb	200	10	LC-MS/MS	2019-11-14	2019-11-22
Imidacloprid	ppb	40.8	5	LC-MS/MS	2019-11-21	2019-11-21
lpconazole	ppb	166	5	LC-MS/MS	2019-11-21	2019-11-21
Isavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	17.0	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	141	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	627	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	2470	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	2360	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	32.4	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	102	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	19PE008836	112	ND	ND	ND
Glyphosate	ppb	19PE008836	82.3	ND	ND	ND
lmidacloprid	ppb	19PE008837	108	ND	ND	ND
lpconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

#### Definitions:

ppb - parts per billion

Detection Limit - Lowest concentration that can be quantitatively reported with confidence

ND - Not Detected above the limit of quantification

Duplicate - Concentration found in repeat sample analysis

Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample

Matrix Blank - A similar-matrix, blank sample is evaluated

Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

## **BRIEF METHOD DESCRIPTION**

## Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

### Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

# Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

# Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)

Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.

https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

### Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

## Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

## Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

## Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

#### Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

## Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

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Report Date: 2019-12-04 Final Report

# South Dakota Agricultural Laboratories has examined the sample of

Limfinite Package Id: 20191113-003

Lab Sample Id: 19PE009039

Customer Sample Id : Trip Blank - AltEn

Sample Description : Trip Blank - AltEn

Date Collected: 2019-11-12

Date Received: 2019-11-13

ANALYTE	UNIT	AS RECEIVED	DETECTION LIMIT	METHOD	DATE OF EXTRACTION	DATE OF ANALYSIS
Acetamprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Azoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Bifenthrin	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Brassinazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Chlorpyrifos-ethyl	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Chlorpyrifos-methyl	ppb	<5	5	GC-MS/MS	2019-11-18	2019-11-22
Clothianidin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Cyfluthrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyhalothrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cypermethrin 1-4	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Cyproconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Deltamethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22
Desthio-Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Difenoconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Dimoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Dinotefuron	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Epoxiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Fluoxastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Glufosinate	ppb	<10	10	LC-MS/MS	2019-11-19	2019-11-22
Glyphosate	ppb	<10	10	LC-MS/MS	2019-11-19	2019-11-22
lmidacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
lpconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
lsavuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Itraconazole	ppb	<5	5	LC-MS/MS	2019-11-21	2019-11-21
Metconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Nitenpyram	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Orysastrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Permethrin 1-2	ppb	ND	5	GC-MS/MS	2019-11-18	2019-11-22

Picoxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Posaconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Propiconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Prothioconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Pyraclostrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Ravuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tebuconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Tetraconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiabendazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiacloprid	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Thiamethoxam	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Trifloxystrobin	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-22
Uniconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21
Voriconazole	ppb	ND	5	LC-MS/MS	2019-11-21	2019-11-21

ANALYTE	UNIT	DUPLICATE	SPIKE RECOVERY	MATRIX BLANK	PROCESS BLANK	INSTRUMENT BLANK
Acetamprid	ppb	19PE008837	96.5	ND	ND	ND
Azoxystrobin	ppb	19PE008838	95.5	ND	ND	ND
Bifenthrin	ppb	19PE008836	111	ND	ND	ND
Brassinazole	ppb	19PE008838	125	ND	ND	ND
Chlorpyrifos-ethyl	ppb	19PE008836	109	ND	ND	ND
Chlorpyrifos-methyl	ppb	19PE008836	110	ND	ND	ND
Clothianidin	ppb	19PE008837	124	ND	ND	ND
Cyfluthrin 1-4	ppb	19PE008836	110	ND	ND	ND
Cyhalothrin 1-2	ppb	19PE008836	110	ND	ND	ND
Cypermethrin 1-4	ppb	19PE008836	108	ND	ND	ND
Cyproconazole	ppb	19PE008838	118	ND	ND	ND
Deltamethrin 1-2	ppb	19PE008836	108	ND	ND	ND
Desthio-Prothioconazole	ppb	19PE008838	119	ND	ND	ND
Difenoconazole	ppb	19PE008838	116	ND	ND	ND
Dimoxystrobin	ppb	19PE008838	123	ND	ND	ND
Dinotefuron	ppb	19PE008837	95.6	ND	ND	ND
Epoxiconazole	ppb	19PE008838	110	ND	ND	ND
Fluconazole	ppb	19PE008838	124	ND	ND	ND
Fluoxastrobin	ppb	19PE008838	103	ND	ND	ND
Glufosinate	ppb	<10	73.5	ND	ND	ND
Glyphosate	ppb	<10	83.7	ND	ND	ND
lmidacloprid	ppb	19PE008837	108	ND	ND	ND
lpconazole	ppb	19PE008838	103	ND	ND	ND
Isavuconazole	ppb	19PE008838	118	ND	ND	ND
Itraconazole	ppb	19PE008838	130	ND	ND	ND
Metconazole	ppb	19PE008838	110	ND	ND	ND
Nitenpyram	ppb	19PE008837	105	ND	ND	ND
Orysastrobin	ppb	19PE008838	108	ND	ND	ND
Permethrin 1-2	ppb	19PE008836	107	ND	ND	ND
Picoxystrobin	ppb	19PE008838	107	ND	ND	ND
Posaconazole	ppb	19PE008838	117	ND	ND	ND
Propiconazole	ppb	19PE008838	116	ND	ND	ND
Prothioconazole	ppb	19PE008838	122	ND	ND	ND

Pyraclostrobin	ppb	19PE008838	105	ND	ND	ND
Ravuconazole	ppb	19PE008838	122	ND	ND	ND
Tebuconazole	ppb	19PE008838	93.5	ND	ND	ND
Tetraconazole	ppb	19PE008838	116	ND	ND	ND
Thiabendazole	ppb	19PE008838	126	ND	ND	ND
Thiacloprid	ppb	19PE008837	91.4	ND	ND	ND
Thiamethoxam	ppb	19PE008837	106	ND	ND	ND
Trifloxystrobin	ppb	19PE008838	96.5	ND	ND	ND
Uniconazole	ppb	19PE008838	122	ND	ND	ND
Voriconazole	ppb	19PE008838	97.9	ND	ND	ND

#### Definitions:

ppb - parts per billion

Detection Limit - Lowest concentration that can be quantitatively reported with confidence

ND - Not Detected above the limit of quantification

Duplicate - Concentration found in repeat sample analysis

Spike Recovery - Recovery based on a known amount of active ingredient spiked into a similar-matrix, blank sample

Matrix Blank - A similar-matrix, blank sample is evaluated

Process Blank - A sample without any matrix (soil, vegetation etc) is processed through the sample analysis procedure Instrument Blank - Injection solvent is run to demonstrate no carryover between injections on the instrument

## **BRIEF METHOD DESCRIPTION**

## Strobins in Water - Purpose and Scope

Strobins are fairly polar and are usually determined by LC-MS/MS. The limits of detection for the strobins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Strobins in Water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

### Strobins in Water - Basic Principles

Strobin water samples are extracted into aqueous methanol followed by filtration and preparation for LC-MS/MS.

### Azoles in water - Purpose and Scope

Azoles are not ionic and are soluble in many organic solvents. Several of them are volatile enough for gas chromatography, but in this laboratory, LC-MS/MS has been used for azole analysis. The limits of detection for the azoles are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Azoles in water - References

Analytical Methods for Pesticides and Plant Growth Regulators. (G. Zweig, ed.) Vol.X, pp. 347 19.1.2.2 Klein and Alder. JAOAC. 86(5): 1015-37 (2003). 19.1.2.3 Ramsteiner et al. JAOAC. 57(1): 192-201 (1974).

# Azoles in water - Basic Principles

Azole water samples can be extracted in aqueous methanol, filtered and prepared for LC-MS/MS analysis.

# Permethrins in water - Purpose and Scope

The pyrethroids are neutral compounds, some of which may contain the cyclopropanecarboxylic ester group and some which do not. Some contain the –CN (cyano) group, and most of the synthetic pyrethroids contain a halogen. As esters, they are susceptible to hydrolysis in basic solution, but not extremely so. They are quite nonpolar, so they are capable of being extracted into a variety of organic solvents. They are usually stable to gas chromatography, so GC-MS/MS will be a common approach to their analysis. Most of these compounds occur as cis and trans isomers, so multiple peaks may be observed. The limits of detection for the permethrins are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Permethrins in water - References

Huang and Pignatello. JAOAC 73(3): 443-446 (1990

A.E. Smith. J. Agric. Food Chem. 29: 111-115 (1981)

Ramsteiner et al. JAOAC 57(1): 192-201 (1974)

Improved LC/MS/MS Pesticide Multiresidue Analysis Using Triggered MRM and Online Dilution.

https://www.agilent.com/cs/library/applications/5991-7193EN.pdf

### Permethrins in water - Basic Principles

Water sample is blended with methanol/water and salt is added. The sample is then extracted with dichloromethane and dried over sodium sulfate. Sample is evaporated and prepared for GC-MS/MS analysis.

## Neonicotinoids in water - Purpose and Scope

Neonicotinoids are a class of neuro-active insecticides chemically similar to nicotine. The limits of detection for the neonicotinoids are 1 ppb for limit of detection and 5 ppb for limit of quantitation.

#### Neonicotinoids in water - References

J. Klein and L. Alder, JAOACI 86(5): 101501037 (2003)

## Neonicotinoids in water - Basic Principles

Neonicotinoids are fairly polar and are extracted with aqueous acetonitrile, filtered and prepared for LC-MS/MS analysis.

## Glyphosate and Glufosinate in water - Purpose and Scope

This method is used for the determination of glyphosate and glufosinate residue in water. The limits of detection for the organophosphates are 3 ppb for limit of detection and 10 ppb for limit of quantitation.

#### Glyphosate and Glufosinate in water - References

P. Alferness and Y. Iwata, J. Agric. Food Chem. 42 (12) 2751-59 (1994) for the derivatization for GC/MS L. Lundgren, J. Agric. Food Chem. 34 535-538 (1986) (DNP derivative)

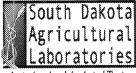
# Glyphosate and Glufosinate in water - Basic Principles

Water sample is filtered and added to the anion exchange resin. Shake water/resin slurry and add to a chromatography column. Glyphosate and Glufosinate is eluted with acidified water, evaporated, reconstituted and derivatized for LC-MS/MS analysis.

Submitted by the customer:

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Seuter Lagger Parker	11/11/2019		WS	U	WW		
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Received by Christina Schepker



# Pesticide Residue Sample Submission Form

South Dakota Agricultural Laboratories 20191113-069 19PE008836-008839 1335 Western Avenue Brookings, SD. 57006 Agricultural and Analytical Testing (605) 692-7325 Atten Washingtoan (Mark Poweis) Zip: 208 98922 Phone: 7 (402) 477 - 2936 \*\*Email: Mark Pornsid CNebrooks 300 \*Sample ID must be marked clearly on the sample you submit. \*\*Results will be emailed to the provided email address. Billing Information & Check box if billing is the same as the customer information States Zio: City:\_\_\_ Email: Individual tests are \$162 each, unless otherwise marked. Scans are \$212 and include all of the compounds in a particular category. Acceptable samples include Vegetation, Water or Soil. Call to confirm other Thank you for choosing South Dakota Agricultural Labs! We do add analytes to our testing regiment throughout the year. If a chemical of interest is not listed, please call us: (605) 692-7325 How much sample should you send? Please send 30g of vegetation or 100g of soil to run an individual test. What does this look like? For vegetation, it would be about a quart sized bag packed full. If more than one test is required, please fill a gailon sized bag. For soil samples, please send 2 cups, if more than one test is required send 4 cups. **Analyses offered** Please turn page over to view the current pesticide analyses. If you are interested in a screen of active ingredients, please check the box next to the bold-faced heading. This will include all active ingredients within the PGR screen for \$212. Example: PGR Screen If you are interested in single analyses, please circle the active ingredients. The cost of each individual analyte is \$162 unless otherwise marked. Example: (Mesotrione) Sample\s received at SD Ag Labs Date <u>2019- II-I S</u>

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·		- S <sub>20</sub> ,	
Azoles 💟	Azoxystrobin	Castaman	Fludioxonii
Brassinazole	Pyraclostrobin	Sulams	Flumioxazin
Cyproconazole	Orysastrobin	Cloransulam	Fluopyram
Difenoconazole	Trifloxystrobin	Florasulam	Fluthiacet methyl
Epoxiconazole	Fluoxastrobin	Flumetsulam	Flutriafol
Fluconazole		Penoxsulam	Fluxapyroxad
Ipconazole	PGR	Pyroxsulam	Fiuaziflop
Isavuconazole	2, 4-D	and a constant of the constant	Fomesafen
ltraconazole	2,4,5-T	OP Scan S \$262	Halosulfuron \$212
Metconazole	2,4,5-TP	Glyphosate \$212	Hexazinone
Posaconazole	2,4-D8	Glufosinate \$212	Indazifiam
Propiconazole	2,4-DP		Indoxacarb
Prothicconazole	Bentazon	Individual Active	lodosulfuron \$212
Prothicconazole	Bromacil	Ingredients	Isoxaflutole
Metabolite 1	Bromoxynil	Abemectin	Lactofen
Prothicconazole	Clopyralid	Acetamiprid	Lincomycin
Metabolite 2	DCPA	Aciflurofen	Linuran
Ravuconazole	Dicamba	Aldicarb	Maisthion
Tebuconazole	Fluroxypyr	Aldicarb suifone	Mandipropamid
Tetraconazole	MCPA	Aldicarb sulfoxide	Mesotrione
Thiabendazole	MCPP	Aminocyclopyrachlor	Methiocarb
Uniconazole	Picloram	Aminopyralid \$212	Methomyl
Voriconazole	Pyrasulfotole	Benzovendiflupry	Nitenpyram
8 a 200	Quinclorac	Bicyclopyrone	Oryzalin
Permethrins 7	Triclopyr	Carbaryl	Oxamyl
		Carbofuran	Oxathiapiprolin
Cyhalothrin 1-2	Pre-Emergents	Carfentrazone	Oxyfluorfen
Permethrin 1-2	Acetochlor	Chlorantraniliprole	Paclobutrazol
Cyhalothrin 1-4	Alachior	Chlorophacinone	Paraquat \$312
Cypermethrin 1-4	Atrazine	Chlorpyrifos-ethyl	Pinoxaden
Deltamethrin 1-2	Dimethenamid	Chlorpyrifos-methyl	Prodiamine
IMI	Metolachlor	Chlorsulfuron \$212	Propachlor
lmazalil	Metribuzin	Clethodim	Propanil
Imazamethabenz	Pendimethalin	Clethodim sulfane	Propazine
Imazamox	Prometon	Clethodim sulfaxide	Propoxur
lmazapic	Simazine	Clomazone	Propoxycarbazone
lmazapyr	SU 💮	Cyanazine	Pydiflumetofen
Imazaquin	Chlorimuron	Diazinon	Pyroxasulfone
Imazethapyr	Formasulfuron	Diffufenzopyr	Quizalofop
some of		Dinotefuran	Saflufenacil
Neonics Z	Mesasulfuron Metsulfuron	Diguat \$312	Sedaxane
Acetamiprid	Nicosulfuron	Dithiopyr	Sulfameturon methyl
Clothianidin	Primisulfuron	Diuron	Sulfentrazone
Thiacloprid	Prosulfuron	Diuron metabolite	Tebuthiuron
Imidacloprid	Rimsulfuron	Ethalfuralin \$212	Tembotrione
Thiamethoxam	Sulfometuron	Emamectin benzoate	Terbacil
Nitenpyram		Fenoxaprop	Thiencarbazone
Dinotefuran	Sulfosulfuron	Fipronil	Thiophanate methyl
Strobins To	Thifensulfuron	Fipronil sulfide	Topramezone \$212
Dimoxystrobin	Triasulfuron	Fipronil sulfone	Trifuralin \$212
Picoxystrobin	Tribenuron Triflusulfuron	Fluazinam	Trimethoprim
•	HHUSURUFOR	Flucarbazone	· · · · · · · · · · · · · · · · · · ·